Reports File

GEORGIA INSTITUTE OF TECHNOLOGY
Engineering Experiment Station

PROJECT INITIATION

Date: February 21, 1974

Project Title: "Dielectric Measurements"

Project No.: A-1606

Project Director: Dr. S. H. Bomar, Jr.

Sponsor: Southern Research Institute

Effective February 8, 1974 Estimated to run until May 8, 1974

Type Agreement: P.O. K 4704 Amount: $ 20,200

Reports Required: Final Report

Sponsor Contact Person (s): Ms. Sylvia A. Puckett
Buyer
Southern Research Institute
2000 Ninth Avenue
South Birmingham, Alabama 35205

Assigned to HIGH TEMPERATURE MATERIALS AND SPECIAL TECHNIQUES Division

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PROJECT TERMINATION

Date: March 20, 1975

Project Title: Dielectric Measurements

Project No.: A-1606

Project Director: Dr. S. H. Bomar, Jr.

Sponsor: Southern Research Institute

Effective Termination Date: November 30, 1974

Clearance of Accounting Charges: March 31, 1975

Grant/Contract Closeout Actions Remaining: None

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A-1606
Final Technical

ENGINEERING EXPERIMENT STATION

GEORGIA INSTITUTE OF TECHNOLOGY • ATLANTA, GEORGIA 30332

March 10, 1975

Southern Research Institute
2000 Ninth Avenue
South Birmingham, Alabama 35205

Attention: Mr. Bill Pittman

Subject: SRI Purchase Order K 4704; Final Technical Report for Dielectric Measurements on Silicon Dioxide Composite Materials (Georgia Tech Project A-1606).

Gentlemen:

In accordance with the subject purchase order, we have accomplished high temperature dielectric measurements on two types of fused silica composite materials supplied by the Philco-Ford Corporation of Newport Beach, California. The specimen materials exhibited a three dimensional woven structure of fibers embedded in an unidentified matrix; it is our understanding that the principal constituent of both the fibers and matrix was amorphous silicon dioxide and that the samples represented two varieties of the composite material known as AS3DX. One group of samples was designated "Type A (untreated)" and the second was designated "Type B (treated)". These designations were preserved throughout our work.

The microwave measurement system and data reduction procedure have been described previously and appropriate references have been supplied to personnel of Lockheed Missiles and Space Company (References 1-4). Briefly, the measurement method involves heating a flat specimen on one side and continuously recording its microwave transmission properties under transient conditions. Temperature data are simultaneously recorded, and computer processing techniques are subsequently employed to reconstruct the dielectric constant and loss tangent versus temperature functions which must have existed to yield the measured transmission coefficient and insertion phase delay. Matrix algebra is used in the data reduction process in such a manner that the final dielectric constant and loss tangent versus temperature functions represent a best fit to information obtained at eight discrete times during the measurement run.

The microwave operating frequency can be either 9.3 GHz (x-band) or 24 GHz (k-band). Specimens are in the form of round disks, six inches in diameter for x-band operation, or three inches in diameter for k-band. A one-inch diameter specimen for thermocouples is needed to accompany each of the microwave samples. Sample thickness is on the order of 0.50 to 0.75 inch, and preferably an integral multiple of one-half wavelength at the operating frequency. Since the dielectric constant and loss tangent of most materials are not strong functions of frequency in the microwave region of the spectrum, the choice of measurement frequency is usually determined by sample size; in this program, a measurement frequency of 24 GHz and a sample diameter of three inches were selected.

The microwave and thermal samples are mounted in a ceramic carrier disk approximately 16 inches in diameter. Three sets of samples (each set consisting of a large microwave specimen and its accompanying small thermal specimen) were mounted in each carrier: (1) a set of Type A (untreated) composite material, (2) a set of Type B (treated) composite material, and (3) a set of slip-cast fused silica specimens to serve as reference material. All the samples on a particular carrier disk are then tested simultaneously.

The carrier disk is enclosed in a furnace structure, and is rotated about its axis at about 20 revolutions per minute. The samples mounted on the carrier are heated on one side by oxyacetylene torches so that a temperature gradient is established; typically the heating period is on the order of three minutes, and during this time a maximum surface temperature of 3500 to 4000° F is attained. The samples do not reach a steady state temperature distribution, so that subsequent data reduction must account for transient conditions.

The furnace is constructed so that each specimen passes through the microwave beam once in each carrier revolution and is exposed to the oxyacetylene flames during the remainder of the carrier revolution. Special care is exercised to avoid the presence of flames in the microwave beam, since ionized gases in the flames interfere with the microwave measurements. The system is designed so that the rotating carrier disk and samples intercept one leg of a continuously operating microwave bridge network; the beam is propagated in free space in the vicinity of the furnace, and focussed to the sample diameter at the sample location so that all losses can be accounted for. A block diagram of the system is shown in Figure 1 and photographs of one of the carriers are shown in Figures 2 through 4.

Measurement runs were performed on January 23 and February 5, 1975. Mr. Bert Ward of Lockheed Missiles and Space Company witnessed the January 23rd test. Measured front surface, rear surface and interior temperatures for all three specimen materials run on January 23rd are shown in Figures 5 through 7.

In order to perform the data reduction, it was necessary to estimate the density, heat capacity, and thermal conductivity of the specimens over the range of temperatures covered by the measurements (room temperature to approximately 3500° F). The density was measured prior to mounting each of the specimens in the carriers. The heat capacity of slip-cast fused silica is known over the temperatures of interest, and the heat capacity of the composite materials was assumed to match that of fused silica since these materials were composed principally of amorphous silica. The thermal

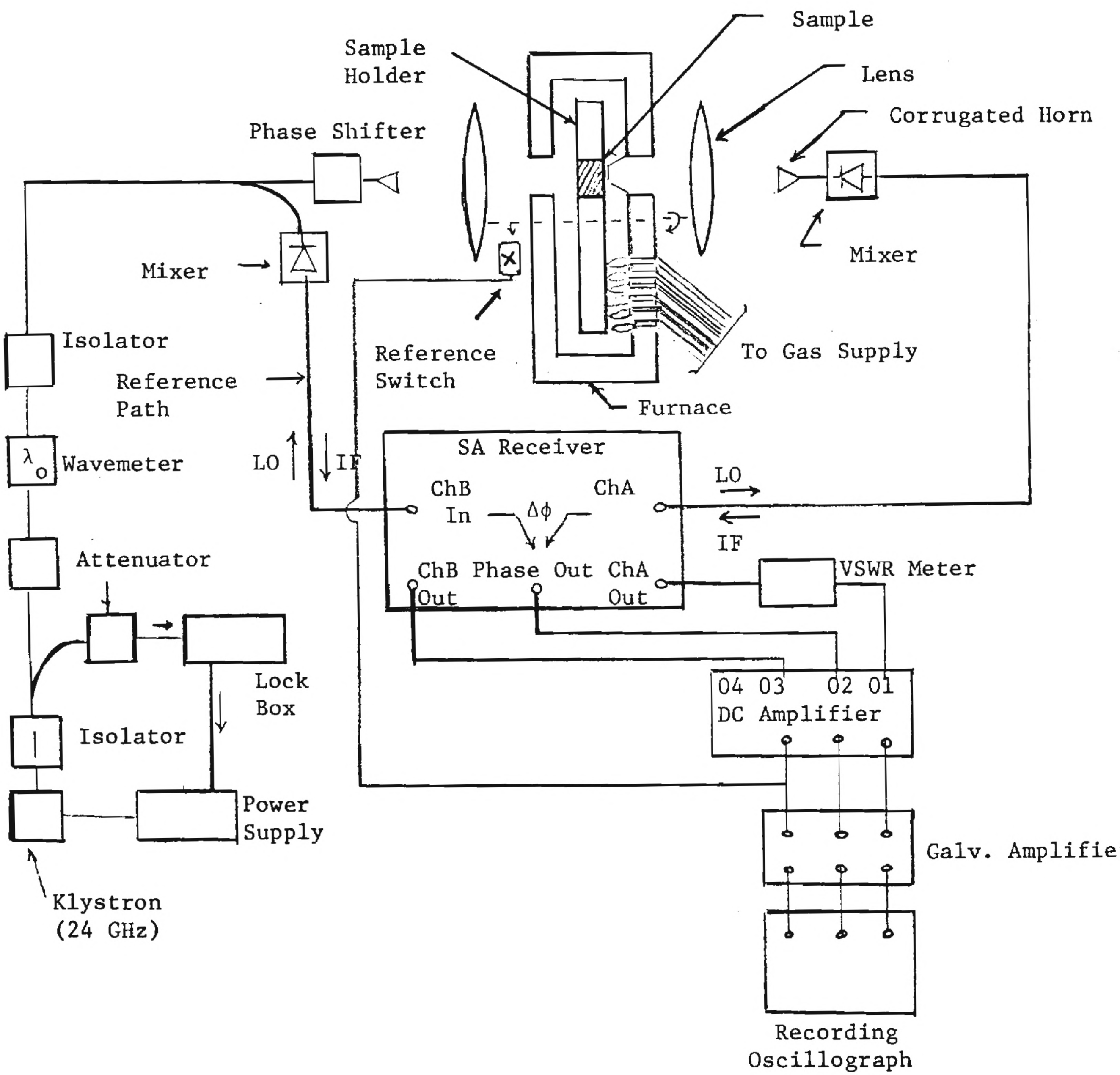

Figure 1. High Temperature Free-Space Focused System Dielectrometer.

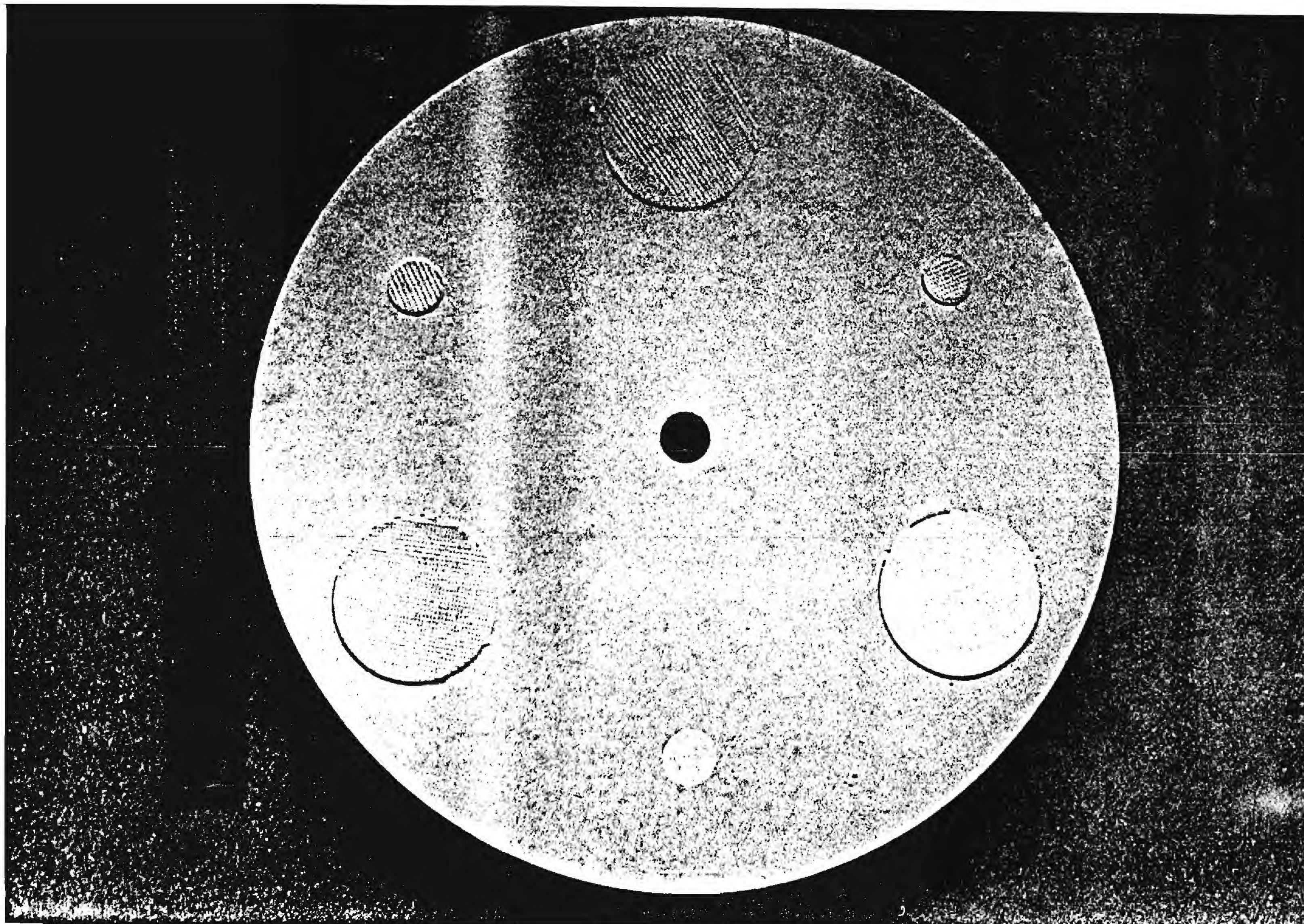

Figure 2. Front Surface of Carrier Disk with Microwave and Thermal Samples.

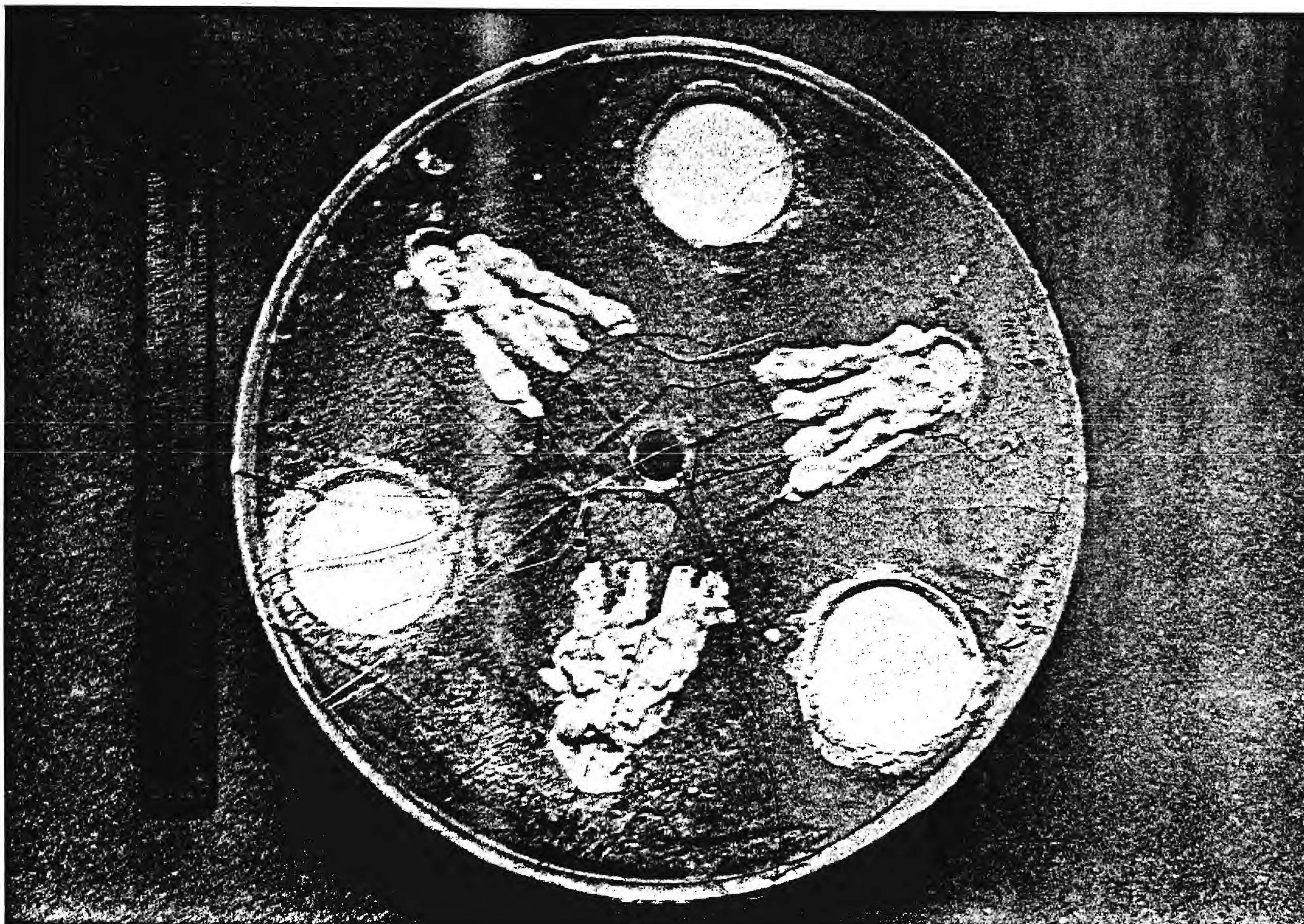

Figure 3. Rear Surface of Carrier Disk with Samples and Thermocouples Installed.

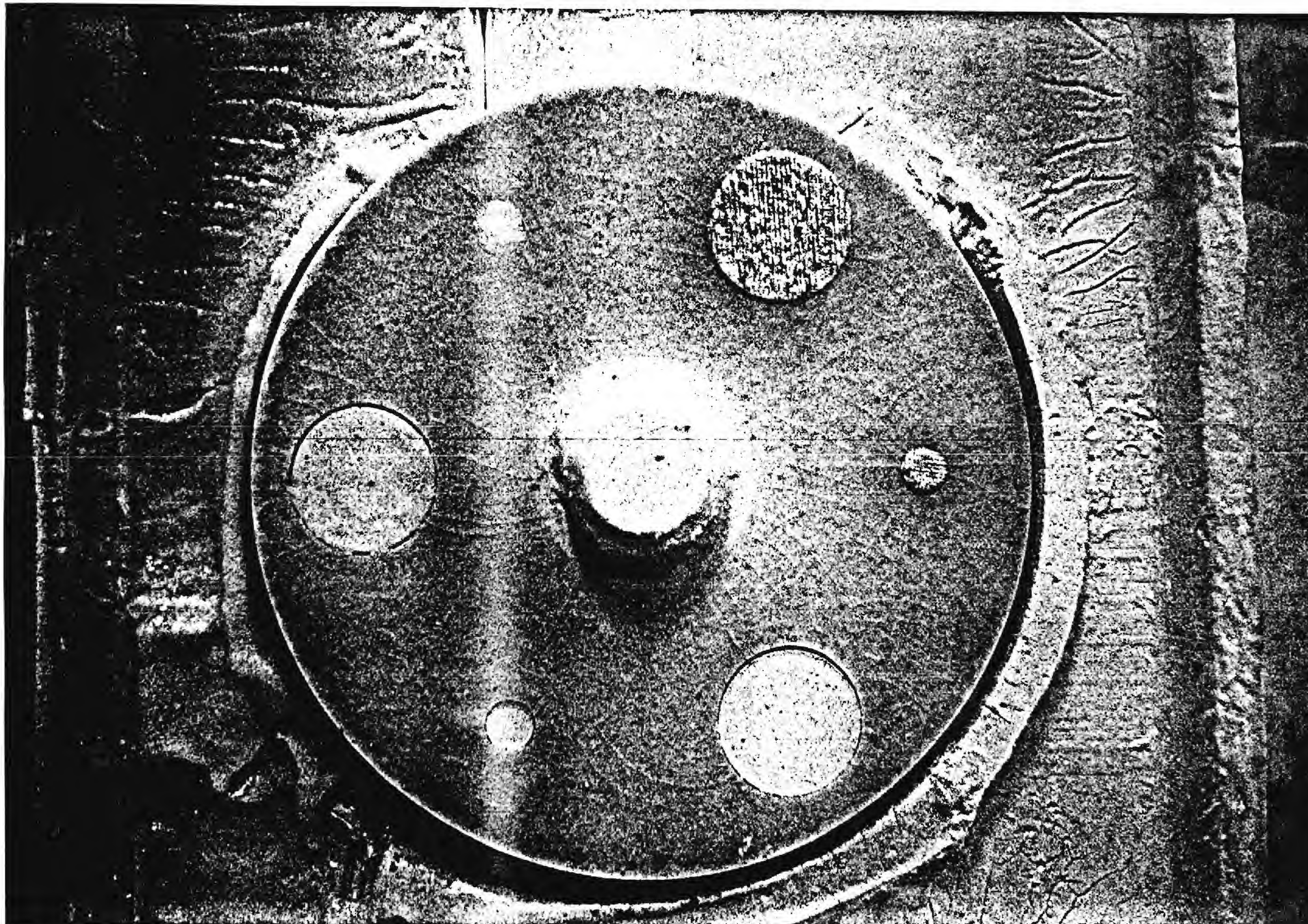

Figure 4. Front Surface of Carrier Disk after Measurement Run (Type B composite material is at upper right).

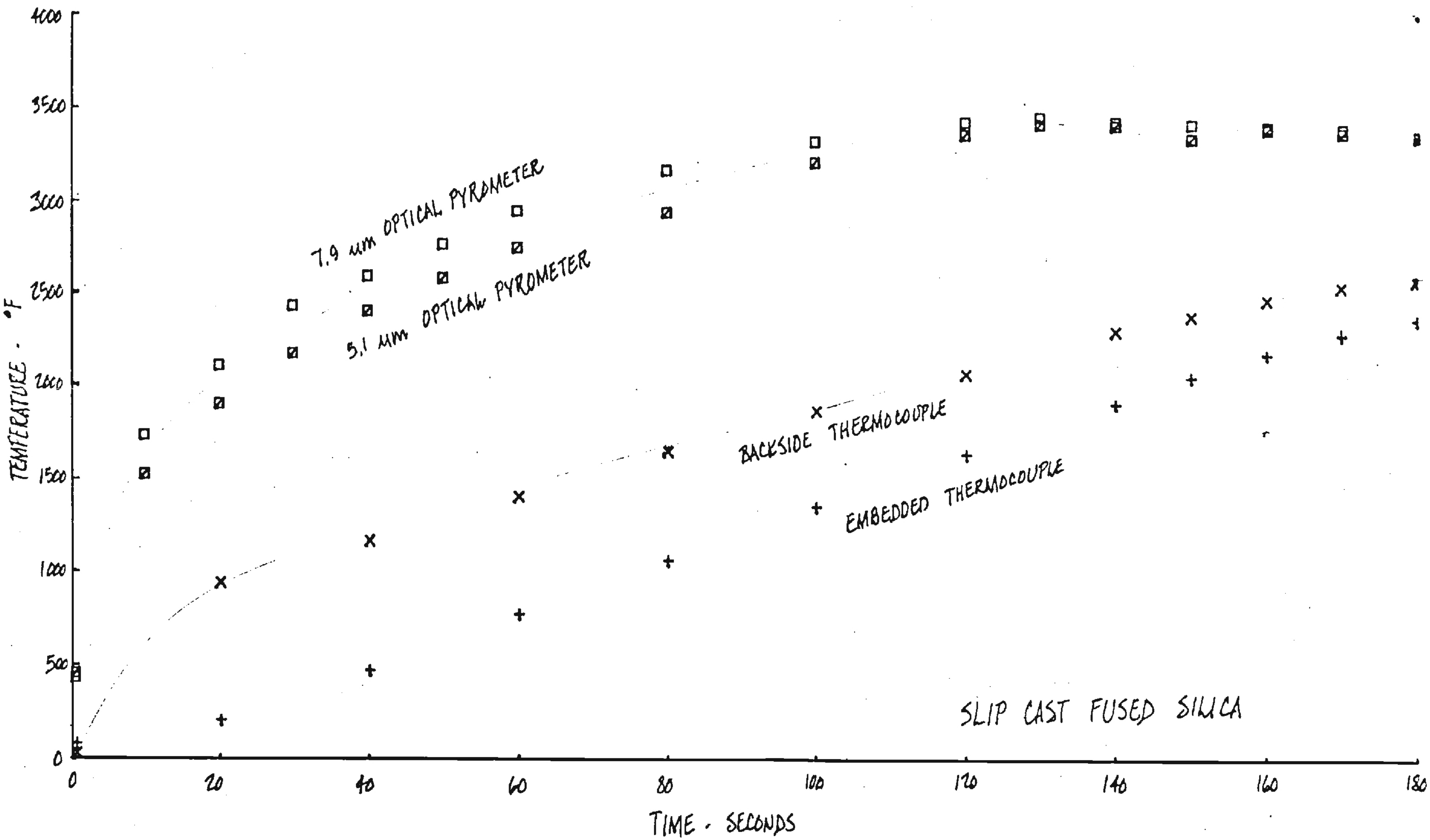

Figure 5. Temperature versus Time Data for Slip-Cast Fused Silica.

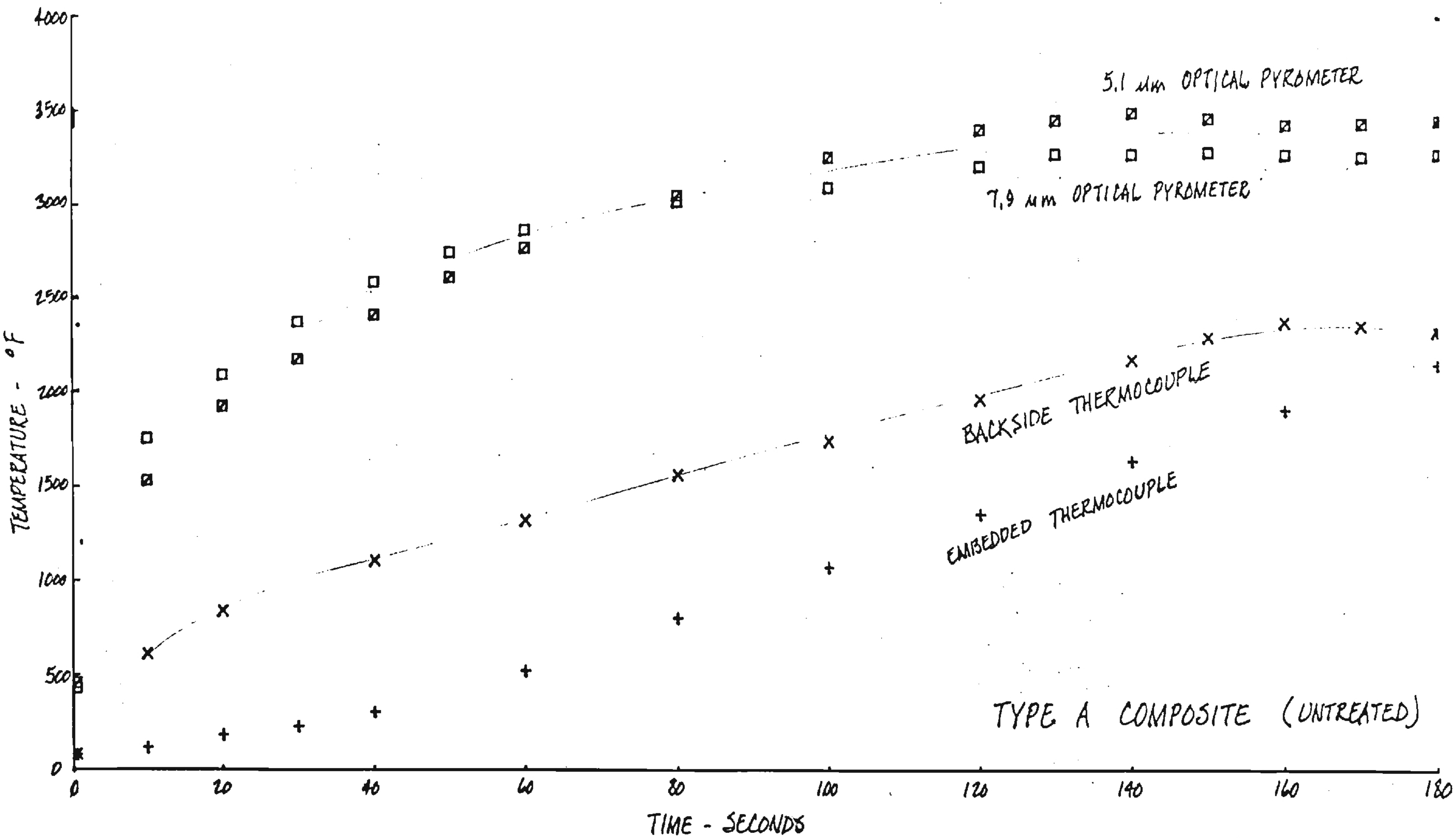

Figure 6. Temperature versus Time Data for Type A Composite.

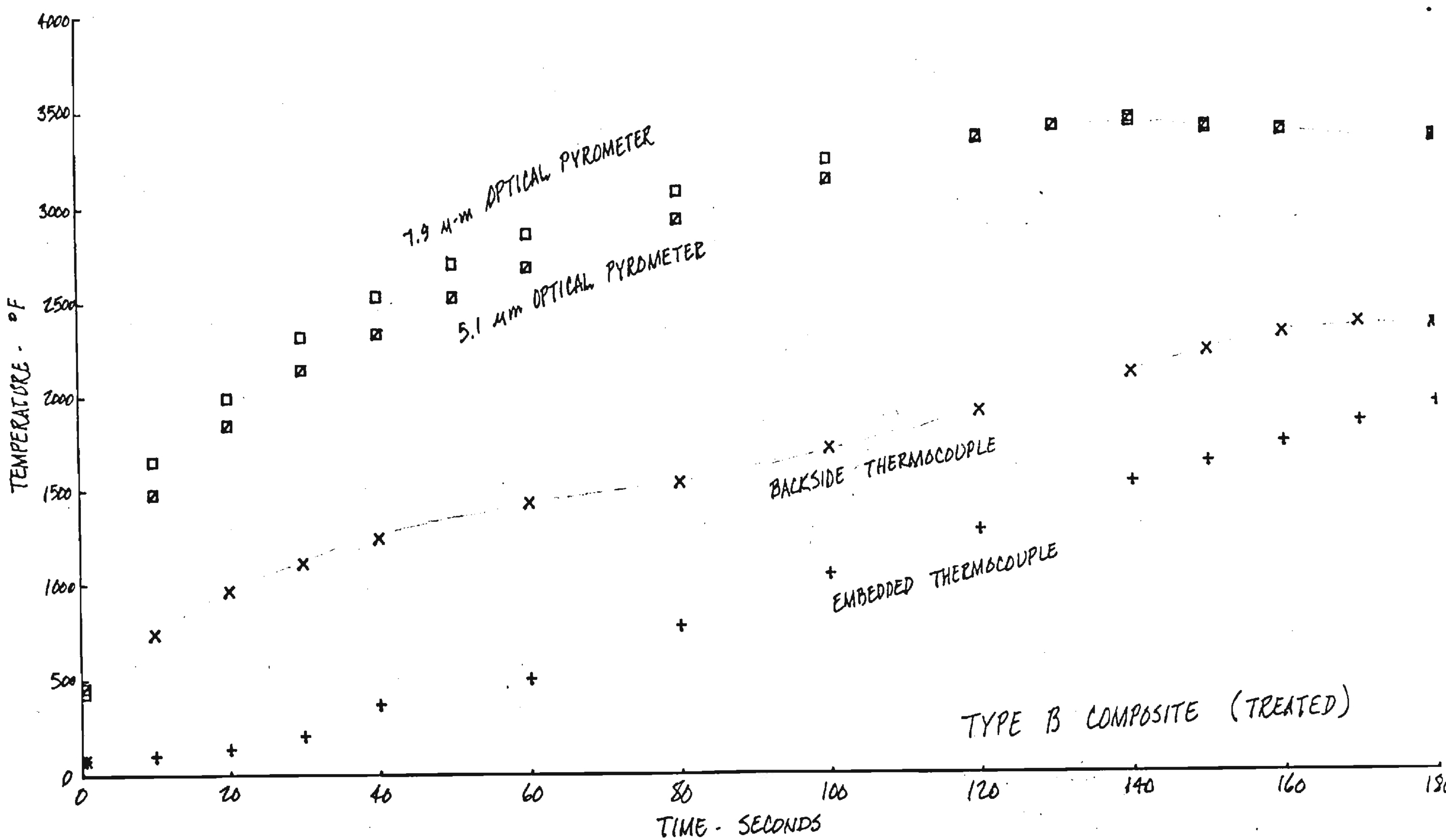

Figure 7. Temperature versus Time Data for Type B Composite.

conductivity of slip-cast fused silica is also known over the temperature range of interest, and this property for the composite material was assumed to be represented by data published by T. M. Place for AS3DX at lower temperatures (Reference 5) and by data for slip-cast fused silica at higher temperatures (Reference 4).

The computer programs used for microwave data reduction require that temperature profiles (temperature versus distance) and sample thicknesses be known as functions of run time. This information is generated by a thermal computer program; the thermal program contains routines which account for sintering in silica based materials in order to calculate thickness, and the sintering functions use an activation energy and a rate constant. These two quantities have been determined for slip-cast fused silica but required estimation by a trial and error procedure for the two composite materials. However, they were determined with sufficient accuracy to successfully predict the final thickness of the composite materials within 0.001 inch.

It is of interest to note that the thicknesses of slip-cast fused silica and Type A composite material were reduced during the measurement run as would be expected. The thickness of Type B composite material increased during the run and a black charred layer formed beneath the specimen surface. It can be presumed that the thickness increase was associated with formation of the charred layer, and this effect offset the shrinkage that should have occurred because of sintering. Specimen densities and initial and final thicknesses are shown in the accompanying table for the two measurement runs.

The final dielectric constant and loss tangent data for the January 23rd run are shown in Figure 8. The plots in Figure 8 for slip-cast fused silica are typical and correlate well with previous measurements. It is normal practice to include slip-cast fused silica on all measurement carriers for reference purposes, and this reproduction of previous results tends to confirm that the measurement equipment and data reduction routines operated correctly, especially if the "unknown" materials should give unexpected results. The plots in Figure 8 for the Type A composite material show that the dielectric constant increased more rapidly than expected over the temperature range of 1000° to 2500° F, but percentagewise the increase is small. Above 2000° F, it is believed that the dielectric constant is dependent on both temperature and time. Thus, one must exercise caution in using these data above 2000° F for purposes of calculation.

The final dielectric data for the Type B composite material were not plotted in Figure 8. The dielectric constant and loss tangent for this material behaved in a manner similar to Type A material up to 2500° F. Above 2500° F, the measured attenuation of the Type B sample was at least 5 dB, so that the dielectric constant calculation was not valid. The sample appeared to be charred at the conclusion of the test, as seen in Figure 4, so that the severe signal attenuation was presumably caused by char buildup.

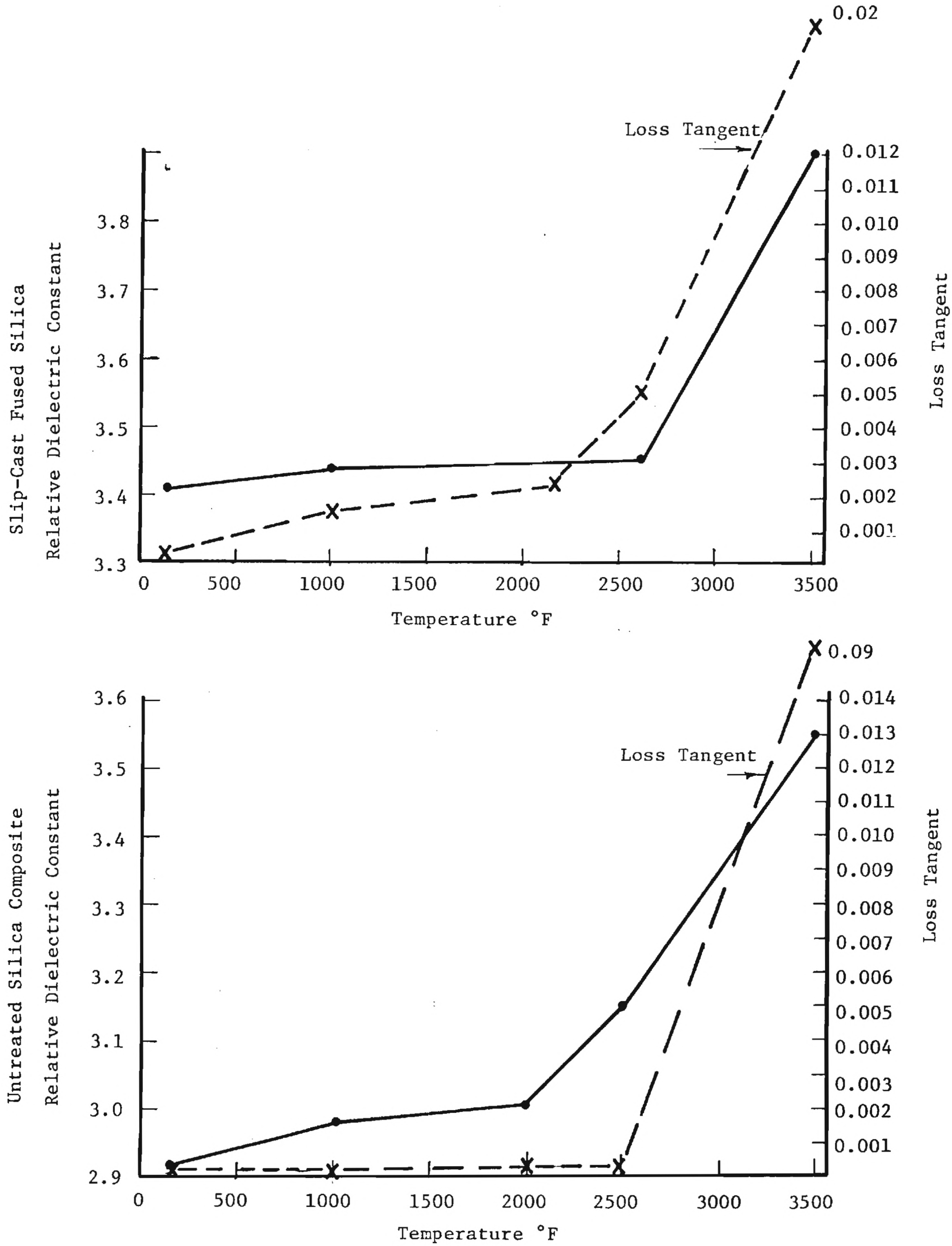

Figure 8. Dielectric Constant and Loss Tangent of Measured Samples at f = 24 GHz.

The February 5th measurement run was terminated at the end of about 60 seconds due to an equipment failure. However, the data were processed and found to be virtually identical to the January 23rd run up through the termination time.

SPECIMEN DENSITIES AND THICKNESSES

	Slip-Cast Fused Silica	Type A Composite	Type B Composite
January 23rd Run:			
Density (gm/cm^3)	1.94	1.71	1.71
Initial Thickness (inch)	0.680	0.857	0.857
Final Thickness (inch)	0.660	0.826	0.912
Maximum Temperature (° F)	3440	3420	3430
February 5th Run:			
Density (gm/cm^3)	1.94	1.69	1.73
Initial Thickness (inch)	0.677	0.858	0.856
Final Thickness (inch)	0.674	0.856	0.854
Maximum Temperature (° F)	1930	1900	1900

References

1. H. L. Bassett and S. H. Bomar, Jr., "Dielectric Constant and Loss Tangent Measurement of High Temperature Electromagnetic Window Materials," Georgia Tech Project A-1089, Final Technical Report AFWL-TR-69-92, Contract F29601-68-C-0060, June 1969.

2. H. L. Bassett and S. H. Bomar, Jr., "High Temperature Complex Permittivity Measurements on Reentry Vehicle Antenna Window Materials," Georgia Tech Project A-1259, Final Technical Report AFWL-TR-71-189, Contract F29601-70-C-0069, January 1972.

3. H. L. Bassett and S. H. Bomar, Jr., "Complex Permittivity Measurements During High Temperature Recycling of Space Shuttle Antenna Window and Dielectric Heat Shield Materials," Georgia Tech Project A-1389, Final Technical Report NASA CR-2302, Contract NAS1-11267, September 1973.

4. S. H. Bomar, Jr., H. L. Bassett, P. R. Theobald and J. M. Newton, "Radome Materials Evaluation," Georgia Tech Project A-1408, Final Technical Report AFAL-TR-73-222, Contract F33615-72-C-1615, July 1973.

5. T. M. Place, "Design Properties for Three Dimensionally Reinforced Silica," Proceedings of the Twelfth Symposium on Electromagnetic Windows, Georgia Institute of Technology, Atlanta, June 1974, pp 47-51.

Respectfully submitted,

Steve H. Bomar, Jr.
Project Director

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